

# इंटरनेट

# मानक

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IS 3025-54 (2003): Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater, Part 54: Nickel [CHD 32: Environmental Protection and Waste Management]



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भारतीय मानक

जल एवं अपशिष्ट जल के नमूने लेना और परीक्षण  
(भौतिक एवं रासायनिक) की विधियाँ

भाग 54 निकिल

( पहला पुनरीक्षण )

*Indian Standard*

METHODS OF SAMPLING AND TEST (PHYSICAL AND  
CHEMICAL) FOR WATER AND WASTEWATER

PART 54 NICKEL

( *First Revision* )

ICS 13.060.50

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**BUREAU OF INDIAN STANDARDS**  
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NEW DELHI 110002

## FOREWORD

This Indian Standard (Part 54) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Pollution caused by substances, on which biotic and abiotic agencies of decomposition are ineffective, is a unique type of pollution. Toxic trace elements and heavy metals come under the category of non-degradable pollutants. The problem caused by these elements is in fact due to their concentration in the environment in the bio-available state and above a certain concentration become harmful to the living organism.

Nickel is not so widely distributed in the earth's crust and the nickel content in the soil is of the order of 100 µg/g. The levels of nickel in sea water is much lower. Nickel is found in almost all human tissues, but essentially of nickel to human being is yet to be proved. Constant exposure with nickel at higher concentrations in aqueous solutions (from their use in the electroplating baths and in the preparation of Ni-catalysts) can lead to allergic sensitization of the skin and what is known as 'Ni dermatitis'. Airborne Ni and nickel compounds (both in the soluble and in the insoluble forms even at the levels of 1 mg/m<sup>3</sup> and 10 mg/m<sup>3</sup> respectively), are highly hazardous and carcinogenic.

Nickel (Ni) is a known carcinogen of respiratory tract. Important sources of Ni are its sulphide and oxide ores. It is the combustion of fossil fuels which contributes the largest amount of Ni to the environment. About 70 000 tons of Ni are discharged into the environment from combustion of fossil fuels alone on global scale. About 0.03-0.12 mg of Ni is present per cubic metres of air of our crowded cities. Estimated daily intake of Ni in human diet averages around 165 mg per day. During the past few years evidence has accumulated that Ni is nutritionally an essential element. It is associated with the synthesis of Vitamin B12.

People occupationally involved with refining and use of Ni are pre-disposed to nasal cancers. Nickel carbonyl was found to be the principal carcinogen. However, it was later concluded from epidermilogical as well as experimental studies that Nickel sulphide could also be responsible for the carcinogen action. Most common toxic effects produced as a sequel to Ni exposure in large amounts include dermatitis and respiratory disorders. Ni inhibits the activity of a number of enzymes such as maleic dehydrogenase, cytochrome oxidase and isocitrate dehydrogenase while its powder or dust is carcinogenic. Nickel carbonyl, formed as a consequence of reaction of Ni with CO is a volatile compound which is the most toxic of all the forms of Ni. It is a carcinogen and half an hour exposure to about 30 ppm could be lethal to human beings. The illness begins with headache, nausea, vomiting and epigastric or chest pain which are accompanied by fever and leucocytosis. More severe cases proceed to pneumonia, respiratory failure and eventually cerebral oedema and death.

The technical committee responsible for the formulation of IS 3025 : 1964 had decided to revise the standard and publish it as separate parts. This standard is one of the different parts under the IS 3025 series of standards.

In the preparation of this standard, considerable assistance is derived from 'Standard Methods for the Examination of Water and Waste Water', 19th Edition-1995, published by the American Public Health Association, Washington, U.S.A., and Vogel's Text Book of Quantitative Inorganic Analysis, 4th Edition, 1978.

Atomic absorption method as specified in this standard is technically equivalent to Method A of ISO 8288 : 1986 'Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric method'.

The composition of the Committee responsible for formulation of this standard is given at Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'.

*Indian Standard***METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER****PART 54 NICKEL***( First Revision )***1 SCOPE**

**1.1** This standard prescribes two methods for the determination of nickel:

- a) Dimethylglyoxime method, and
- b) Atomic absorption method.

**1.2** Depending upon the concentration range and interference levels, choice of the method is made.

**2 REFERENCES**

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

<i>IS No.</i>	<i>Title</i>
3025 (Part 1) : 1987	Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling ( <i>first revision</i> )
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents, Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents, Part 2

**3 TERMINOLOGY**

For the purpose of this standard, definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

**4 SAMPLING AND STORAGE**

The sampling shall be done as prescribed in IS 3025 (Part 1). The sampling bottles shall be cleaned thoroughly with dilute nitric acid (6N), prior to the final rinsing with water. The water samples should be collected and stored preferably in polypropylene bottles or chemically resistant glass containers. The analysis of such samples is to be carried out within 24 h of sampling. For the preservation, the samples should be acidified with concentrated nitric acid (2 ml of conc nitric acid in 1 litre sample, just to bring down the pH below 2). The acidified samples can be stored for a few days (up to 5 days) in a refrigerator.

**5 PURITY OF THE REAGENTS**

Unless specified otherwise, only pure chemicals and nickel free distilled water shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

**6 DIMETHYLGLYOXIME METHOD****6.1 Principle**

Nickel ions react in presence of oxidizing agents (such as bromine), with dimethylglyoxime to give a soluble red coloured complex containing Ni in higher oxidation states of (III) and (IV). This complex absorbs at 445 nm. This method is applicable in the range of 30 to 500 µg/l of nickel.

**6.2 Interference**

Cobalt and copper interfere in estimation of nickel. Interference of Cu (II), Co (II) and other metals is removed by separation of nickel. [Extraction of Ni (II) dimethylglyoxime in  $\text{CHCl}_3$ , and washing the  $\text{CHCl}_3$  layer with dilute  $\text{NH}_4\text{OH}$ , and re-transferring the Ni (II) to aqueous phase.]

**6.3 Apparatus**

**6.3.1 Spectrophotometer** — for use at 445 nm.

**6.3.2 Standard Volumetric Glasswares**

**6.3.3 pH Meter**

**6.4 Reagents**

**6.4.1 Ammonium Hydroxide** — concentrated (14 N) and 0.5 N.

**6.4.2 Hydrochloric Acid** — concentrated (11 N) and 0.5 N.

**6.4.3 Citric Acid** — 10 percent w/v in water.

**6.4.4 Dimethylglyoxime** — 1 percent w/v in ethyl alcohol.

**6.4.5 Ethyl Alcohol** — 95 percent (v/v).

**6.4.6 Chloroform**

**6.4.7 Bromine Water** — Saturated water with bromine.

#### 6.4.8 Standard Nickel Solution

Dissolve 0.673 g of ammonium nickel sulphate [ $\text{NiSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ], in a small quantity of water, and transfer quantitatively into a 1 000 ml volumetric flask. Dilute upto the mark with water (1.0 ml = 100  $\mu\text{g}$  of Ni).

### 6.5 Procedure

#### 6.5.1 Calibration Curve

Pipette out appropriate portions of standard nickel solution into 100 ml volumetric flasks to contain from 50 to 250  $\mu\text{g}$  of Ni. For the reagent blank pipette out 10 ml of water into a separate 100 ml volumetric flask. To each flask, add 5 ml of 0.5N HCl, 2 ml of bromine water, and 2 ml of conc ammonium hydroxide. Cool to room temperature, add 1 ml of dimethylglyoxime reagent and dilute up to the mark with water. Measure the absorbance at 445 nm after 5 min. Use a reagent blank prepared in an identical manner, using 10 ml of water. Construct a calibration curve by plotting absorbance values against micrograms of nickel in 100 ml of the final solution.

#### 6.5.2 Digestion with $\text{H}_2\text{SO}_4$ - $\text{HNO}_3$

Transfer a suitable volume of homogenized sample to a beaker. (Sample volume depends upon the expected Ni content. If the expected Ni concentration is of the order of 1 mg/l, the sample volume will be about 800 ml. If it is between 10 to 100 mg/l, about 100 ml of the sample will be sufficient.) For preparation of the reagent blank measure equal volume of water in a separate beaker. Add 5 ml of conc  $\text{HNO}_3$  and a few porcelain pieces or glass beads. Heat to boil the solutions, and concentrate carefully on a hot plate to lowest possible volume, cool the solution and transfer quantitatively, to a beaker of smaller size (say 150 ml beaker). Add 5 ml of conc  $\text{HNO}_3$  and 10 ml of conc  $\text{H}_2\text{SO}_4$ . Heat up to the evolution of dense white fumes of  $\text{SO}_3$ . If the solution are not clear add 5 ml of  $\text{HNO}_3$  and reheat until a clear solution (no evolution of brown fumes) results. Cool and transfer quantitatively into a 100 ml volumetric flask. Dilute upto the mark with water, and mix well. Use this solution for the determination of nickel as described in 6.5.3.

#### 6.5.3 Determination of Nickel

Pipette out a portion of the acid digestion solution (containing from 50 to 250  $\mu\text{g}$  Ni) into a beaker. Add 5 ml citric acid solution. Neutralize with conc ammonia solution and add a few drops in excess ( $\text{pH} > 7.5$  ensure with a pH meter or the pH paper of appropriate pH range). Add 2 ml of dimethylglyoxime solution (if Cu or Co is present, more of reagent, about 5 ml is added). Transfer quantitatively into a separatory

funnel and extract the Ni-dimethylglyoxime complex with three 5 ml portions of chloroform. Shake the combined chloroform extract with 10 ml of 0.5 N ammonia solution. Separate the two layers. Collect the ammonia washings into another separatory funnel and shake the ammonia washings with 2 ml of chloroform. Transfer this chloroform layer to the main chloroform layer, and discard the aqueous-ammonia phase. Nickel present in the chloroform phase is returned back to an aqueous phase, by shaking the chloroform layer with two 5 ml portions of 0.5N HCl. Transfer quantitatively the hydrochloric acid layer into a 100 ml volumetric flask. Add about 10 ml of water, 2 ml of saturated bromine water and 2 ml of conc ammonia solution to it. Cool to room temperature, add 1 ml of dimethylglyoxime solution and dilute to 100 ml with water. Measure the absorbance of the nickel complex at 445 nm after 5 min against the reagent blank (prepared in an identical manner using water in place of the sample solution).

### 6.6 Calculation

$$\text{mg Ni/l} = \frac{\mu\text{g of Ni (in 100 ml of the final solution)}}{V_1 \times V_2} \times 100$$

where

$V_1$  = volume in ml of the sample taken for the acid digestion, and

$V_2$  = volume in ml of the digestion solution taken for the final Ni determination.

## 7 ATOMIC ABSORPTION METHOD (DIRECT)

### 7.1 Principle

The nickel content of the sample is determined by atomic absorption spectrophotometry. For dissolved nickel the filtered sample may be directly aspirated to the atomizer. For total recoverable nickel,  $\text{HNO}_3$ - $\text{H}_2\text{SO}_4$  digestion is to be carried out prior to aspiration of the sample. This method is applicable in the range from 0.3 to 10 mg/l. However, the concentration range will vary with the sensitivity of the instrument used.

### 7.2 Interferences

The presence of high concentration of iron and chromium may increase the nickel signal. Most interference may be eliminated by the use of the nitrous oxide acetylene flame but nickel sensitivity will be lowered.

### 7.3 Apparatus

**7.3.1** Atomic absorption spectrophotometer with air-acetylene flames. Hollow-cathode lamp or electrodeless discharge lamp for use at 232.0 nm.

## 7.4 Reagents

**7.4.1 Hydrochloric Acid** — concentrated (11 N), 1:1 and 10 percent v/v.

**7.4.2 Nitric Acid** — concentrated (16 N) and dilute (1 : 499).

**7.4.3 Sulphuric Acid** — concentrated (36 N).

### 7.4.4 Stock Nickel Solution

Dissolve 1.273 g of nickel oxide in a minimum quantity of 10 percent (v/v) HCl and transfer quantitatively into a 1 000 volumetric flask. Dilute upto the mark with water (1 ml = 1.0 mg of Ni).

### 7.4.5 Standard Nickel Solution

Pipette out 10.0 ml of stock nickel solution into a 1 000 ml volumetric flask and make upto mark with water (1.0 ml = 10 µg of Ni).

## 7.5 Procedure

### 7.5.1 Calibration Curve

Prepare standard solutions containing 0 to 60 µg/l of nickel by diluting suitable volumes of standard nickel solution with nitric acid (1 : 499) to 100 ml in volumetric flasks. Prepare a reagent blank in an identical manner using 10 ml of water. Aspirate the reagent blank and carry out zero adjustment. Aspirate sequentially the standard solutions and measure the absorbance at 232.0 nm.

**7.5.2** Construct a calibration curve by plotting absorbance values against micrograms of Ni in 100 ml of the final volume. Ensure that the calibration curve is linear by making the necessary changes in the volume of the standard solution used.

### 7.5.3 Determination of Nickel

For the determination of dissolved nickel content, filtration through 0.45 µm membrane filter, at the

time of sampling, is required. For total recoverable nickel, HNO<sub>3</sub>-H<sub>2</sub>SO<sub>4</sub> digestion is to be carried out (see 6.5.2).

**7.5.4** Add 0.5 ml of nitric acid to a suitable volume of the sample taken (or the solution obtained after digestion which contains 50 to 60 µg of nickel) in a 100 ml volumetric flask. Make up to the mark. Rinse the nebulizer by aspirating water containing 1.5 ml of conc HNO<sub>3</sub>/l. Aspirate the reagent blank and carry out zero adjustment. Aspirate the sample solution and measure the absorbance at 232.0 nm. Determine micrograms of nickel in the solution from the absorbance reading, by referring to the calibration curve.

## 7.6 Calculations

**7.6.1 Soluble Nickel** (direct determination without the digestion step)

$$\text{mg Ni/l} = \frac{\mu\text{g of Ni (in 100 ml of the final solution)}}{V} \times 100$$

where

$V$  = volume in ml, of the sample used.

**7.6.2 Total Nickel** (when digestion is carried out)

$$\text{mg Ni/l} = \frac{\mu\text{g of Ni (in 100 ml of the final solution)}}{V_1 \times V_2} \times 100$$

where

$V_1$  = volume in ml of the sample used, and

$V_2$  = total volume in ml of digested solution used for Ni estimation.

### 7.6.3 Precision and Accuracy

The relative standard deviation reported in the literature for the Ni at 4.0 mg/l concentration level is 9.8 percent.



## ANNEX A

### (Foreword)

#### COMMITTEE COMPOSITION

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### Amendments Issued Since Publication

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